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**ANALYSIS OF A PISTON EXPERIENCING
ENVIRONMENTALLY-ASSISTED CRACKING
AS A RESULT OF COMPRESSIVE OVERLOADING**

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| 13. ABSTRACT (Maximum 200 words) A piston used in service in a closed-ended pressure vessel, as a component of the seal for the vessel, cracked after as few as two high-pressure loading cycles. As a further complication, the finite element analysis was predicting compression on the cracked piston face during the loading cycle. The cracks in the piston emanated in a radial fashion and were measured at 1.5-mm deep. Upon subsequent loading, the cracks grew in length (in large incremental advances), but did not propagate any deeper than the initial 1.5-mm. In all instances the robust design did not leak. A closer look at the finite element analysis revealed that the piston, made from Maraging 200 steel, was loaded in compression to above the compressive yield point of the material. The initial loading set up a residual tensile stress as a result of the compressive yielding. On the next successive loading, this tensile residual stress field, coupled with the hydrogen-rich products in the pressure vessel and a highly susceptible material, resulted in cracking. The fact that the cracks were only 1.5-mm deep suggests that this was the extent of the compressive yielding, and that the cracks arrested themselves once the neutral axis or zero stress was encountered. The fracture morphology of the cracks revealed that they were intergranular in nature, and likely to be the result of hydrogen-induced cracking. At this point in the program the design of the piston was firmly established, so the only alternatives were to prevent the hydrogen from getting to the susceptible material, or to change to a less susceptible material. Consideration of the second alternative revealed no material that possessed the unique combinations of strength, toughness, hydrogen resistance, or producibility that were required. Therefore, plating of the piston with various nickel and chromium combinations was investigated. Laboratory tests were devised using a modified bolt-loaded compact specimen for conducting plating studies. The specimens were loaded near the yield strength of the material and exposed to a hydrogen-rich environment. After over 300 hours of exposure, no cracking had occurred in any of the electroplated nickel specimens. These results were then applied to the piston and placed into service. The piston currently has over 700 high-pressure loading cycles with no cracks observed. | | | | | | | |
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BACKGROUND

In August 1997, testing was conducted of a piston utilized in the seal of a closed-ended pressure vessel. Testing included 100 loading cycles, at pressures ranging from 69 MPa to 414 MPa. Upon completion of the test, the component was disassembled, cleaned, and inspected. Although no pressure losses were observed during the test, it was noticed that cracking of the Maraging 200 steel piston had occurred (Figure 1). Cracking was not observed during testing because cleaning of the system was strictly forbidden. The cracking pattern emanated in a radial fashion around the piston, yet none of the cracks broke through the thickness of the piston. Cracks ranged in length from barely noticeable, to approximately 20-mm in length. Cracking was also observed at many locations approximately half the distance between the center of the piston and the outside diameter. In some cases, cracks that started in the center of the piston appeared to "link up" with the cracks that started at the midpoint between the center and the outside diameter. There was also a characteristic ripple where the cracked surfaces protruded upward, producing an uneven texture on the top surface of the piston. The crack pattern appeared relatively evenly spaced, with a crack spacing of approximately 5-mm to 9-mm.

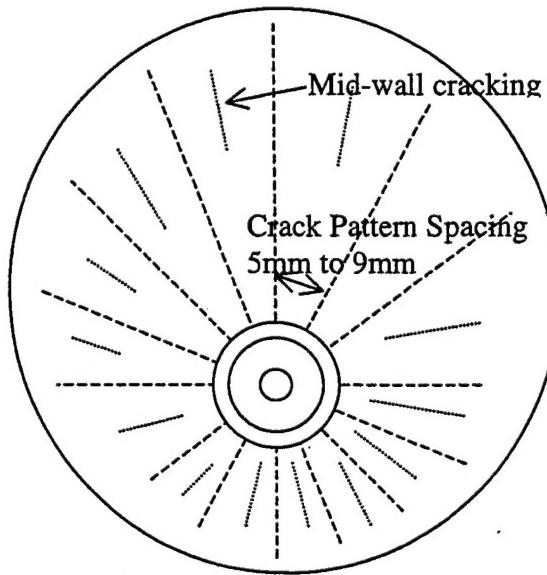


Figure 1. Radial cracking pattern on piston face.

Later in August 1997, testing resumed with the same piston. However, this time cleaning of the surface was allowed for inspection purposes. The sequence of testing consisted of 112 loading cycles ranging from 69 MPa to 414 MPa. Three cracks were chosen and monitored for overall crack length. One of the cracks did not grow in length during the entire 112-round sequence, the second crack extended in length by slightly over 1-mm, and the third crack extended over 3-mm in length. The crack extension observed at the surface did not show a constant crack growth rate, and the cracks grew essentially their entire length increment in only a few loading cycles.

A closer look at the finite element model indicated that the piston did deform into a "dish-like" shape during the loading process. The resultant stresses were compressive on the top surface, exceeding the compressive yield strength of the material, and were predicted to be approximately 1600 MPa.

PISTON EVALUATION

Vendor Certification/Chemistry

The material chemistries as supplied by the vendor, along with the required allowable ranges, are shown in Table 1. All elements are within the required specified limits.

Table 1. Chemical Analysis

| Element | Vendor Chemistry (wt. %) | Required Minimum (wt. %) | Required Maximum (wt. %) |
|---------|-----------------------------|-----------------------------|-----------------------------|
| Al | 0.090 | 0.05 | 0.15 |
| C | 0.003 | | 0.03 |
| Co | 8.400 | 8.00 | 9.00 |
| Mn | 0.020 | | 0.12 |
| Mo | 3.250 | 3.00 | 3.50 |
| Ni | 18.700 | 17.00 | 19.00 |
| P | 0.004 | | 0.01 |
| Si | 0.010 | | 0.12 |
| S | 0.002 | | 0.01 |
| Ti | 0.200 | 0.15 | 0.25 |
| Fe | Balance | | |

Metallography

Metallographic inspection of the cracks revealed an irregular, jagged crack path indicative of intergranular cracking. This cracking is the same type of cracking observed in Maraging 200 bolt-loaded compact specimens that were subjected to a hydrogen-rich environment under a sustained tensile load (ref 1). The material also exhibited a martensitic microstructure, which is typical of a properly heat-treated Maraging 200 steel. Post-test hardness measurements were R_c 43/44.

Material Properties

Tensile specimens were machined from the cracked piston in the C-R orientation, per ASTM Standard E8, and bend specimens were utilized for measuring fracture toughness according to ASTM Standard E813. Testing results are shown in Table 2. Material properties were repeatable and indicative of a good quality Maraging 200 steel. The strength properties, previously reported hardness measurements, and microstructural investigation were all in agreement, and indicate that the heat treatment performed was correct.

Table 2. Mechanical and Toughness Properties

| 0.2% Yield Strength (MPa) | Ultimate Tensile Strength (MPa) | Elongation (%) | Reduction in Area (%) | K _J (MPa \sqrt{m}) |
|---------------------------|---------------------------------|----------------|-----------------------|----------------------------------|
| 1379 | 1441 | 19.8 | 71.8 | 102.1 |
| 1379 | 1427 | 17.0 | 68.0 | 113.2 |
| 1393 | 1448 | 18.0 | 67.5 | 119.9 |

NDI/Inspection

Ultrasonic inspection of the piston indicated that most of the cracks ranged in depth from approximately 1.0-mm to 1.5-mm, with the maximum depth of each crack occurring at the center of the piston. Magnetic particle inspection of the piston revealed that none of the cracks extended to the outside diameter of the piston. Inspection of the top surface revealed a "dishing" effect. The top surface of the piston was 0.05-mm higher on the outside diameter than in the center. The backside of the piston was also plastically deformed and dished. The center of the piston was 0.025-mm higher than the outside edge of the back of the piston.

Fractography/Scanning Electron Microphotography

Two cracks were sectioned from the piston and investigated with scanning electron microphotography (SEM) (ref 2). The first crack initiated at the center of the top surface, and the second crack initiated at the mid-wall location of the top surface. There was some indication of rubbing of the mating surfaces of the cracks, and debris from the products of the pressure vessel were forced into the cracks.

After a thorough cleaning of the surfaces (with plastic replicas), some portions were exposed near the tip of the cracks. Figure 2 is at the tip of the first crack and clearly shows intergranular cracking and the post-fracture ductile tearing that occurred when the crack was sectioned. The second crack seemed similar to the first in fracture appearance, and again intergranular cracking was the predominate fracture morphology. The logical conclusion is that the cracks were not driven by mechanical fatigue loading, but rather the result of hydrogen-induced cracking.

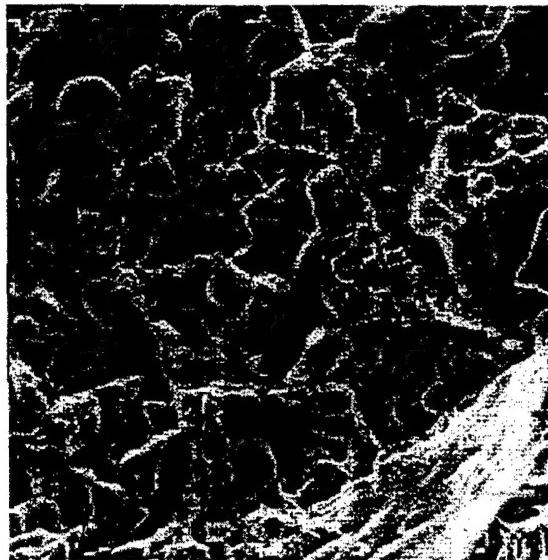


Figure 2. Crack tip morphology showing intergranular cracking.

X-Ray Residual Stress Measurement

X-ray residual stress measurements (ref 3) were taken at six locations on the top surface of the piston. Both hoop and radial residual stress measurements were taken at each of the six locations; the results are shown in Table 3. Note that some of the residual hoop stresses on the top surface approach the yield strength of the material, and that all residual stresses measured indicate tension on the top surface of the piston. Some of the stresses are significantly less than the yield stress of the material, because these measurements bridged across an existing crack, which relieved a significant amount of residual stress. In order for tensile residual stresses of this magnitude to be present, applied compressive stresses must be on the order of two times the yield strength of the material.

Table 3. Residual Stress Measurements

| Location | Hoop Stress (MPa) | Radial Stress (MPa) |
|----------|----------------------|------------------------|
| 1 | +1348 | +875 |
| 2 | +1375 | +920 |
| 3 | +588 | +678 |
| 4 | +1189 | +881 |
| 5 | +878 | +297 |
| 6 | +839 | +254 |

Investigation Conclusions

The three necessary conditions for hydrogen cracking are:

- A source of hydrogen
- A susceptible material
- A sustained tensile loading

In this case, the hydrogen is contained in the pressure vessel contents and cannot be avoided. The sustained tensile stress is a result of compressive yielding, and is inherent in the design, which at this point could not be changed. The Maraging 200 material is a highly susceptible material that is prone to hydrogen embrittlement. All of these conclusions are self-consistent, and suggest the most likely mechanism of failure is hydrogen-induced cracking.

These conclusions also suggest that a suitable method to prevent hydrogen cracking was to create a barrier to prevent the hydrogen from contacting the highly susceptible, highly stressed base material.

MODIFIED BOLT-LOADED COMPACT SPECIMEN

Classic work in the field of preventing hydrogen-induced cracking suggests that one of the best barriers to prevent cracking is the use of a nickel coating. Song and Pyun (ref 4) have stated that, "Hydrogen transport across electrodeposited metals such as Ni has assumed significance in association with the delay of hydrogen induced cracking." As far back as the 1960s, Matsushima and Uhlig (ref 5) noticed that, "Ni coatings alone are protective... resulting in less occlusion of H by steel."

Our approach included modification of the bolt-loaded compact specimen currently being considered for addition to ASTM Standard 1681, "Determining a Threshold Stress Intensity Factor for Environment-Assisted Cracking of Metallic Materials Under Constant Load." Since the piston does not have any sharp stress risers, we felt that the inclusion of a sharp precrack into the specimen would not adequately model this geometry. Therefore, we opted for the inclusion of a 3/8-inch diameter through hole (Figure 3). The base materials chosen for the testing were Maraging 200 and an A723 Grade 2 pressure vessel quality steel, both heat treated to yield strength of 1300 MPa. These materials were chosen because of their unique combination of strength and toughness that was required for the component. Nickel-based alloys would have been the first choice for resistance to hydrogen, however none possessed the required strength. The hydrogen barrier coatings investigated and their thicknesses are included in Table 4.

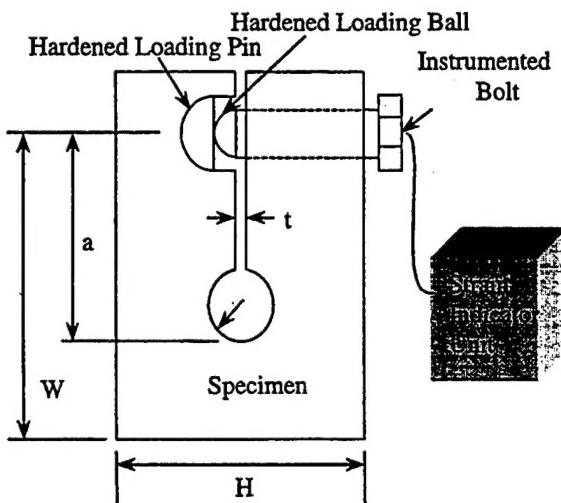


Figure 3. Setup and configuration for the modified bolt-loaded compact testing.

Table 4. Test Matrix of Hydrogen Barrier Coatings and Base Materials

| Base Material | Hydrogen Barrier Coating | Coating Thickness (mm) |
|---------------|-----------------------------|------------------------|
| Maraging 200 | None | |
| | Electroplated Ni | 0.05 |
| | Electroless Ni | 0.05 |
| | Chromium | 0.05 |
| | Electroplated Ni + Chromium | 0.05 |
| | Electroless Ni + Chromium | 0.05 |
| | None | |
| A723 | Electroplated Ni | 0.05 |
| | Electroless Ni | 0.05 |
| | Chromium | 0.05 |
| | Electroplated Ni + Chromium | 0.05 |
| | Electroless Ni + Chromium | 0.05 |
| | None | |

Loading the specimen with the instrumented bolt to a predetermined stress level runs the test. In this case, a bolt load of 31 kN resulted in an applied stress at the process zone of 1200 MPa, or approximately 92% of the yield stress of the base material. This sustained stress acts in the same fashion as the residual stress field in the actual piston. In order to supply the hydrogen to the specimen, an electrolyte of 50% H₂SO₄ and 50% H₃PO₄ by volume, is immediately applied to the process zone and the bolt load is monitored. It is imperative that the electrolyte be applied before loading in order to prevent any oxide from forming and artificially "protecting" the specimen from cracking. Since this type of test is a constant displacement test, as the crack grows, the load will shed. The resistance provided by the coatings will be reflected in the incubation period necessary for the cracking to start. The test duration is arbitrarily set at a maximum exposure time of 300 hours. If cracking has not begun within this time, the test will be halted and the coating assumed to be highly resistant to hydrogen attack. However, if cracking has started, the remainder of the test can be utilized to measure crack growth rates and threshold stress intensity of the base material. These topics will be covered in a separate publication.

TEST RESULTS

Maraging 200

Figure 4 clearly highlights the resistance that each of the coatings provides to the Maraging 200 base material. We can see that the Maraging 200 material with no coating provided approximately one hour incubation time, followed by a decreasing load as monitored by the instrumented bolt. After approximately 250 hours of exposure, the cracking had reached an apparent threshold value, and no further decrease in load was observed. The specimen with the electroless nickel (E-Ni) protective coating provided approximately 30 hours of protection, and rapid cracking of the underlying base material resulted. In this test, after approximately 150 hours of exposure, the cracking appeared to arrest. Cracking immediately resumed after the electrolyte was changed. We believe that the electrolyte had become depleted, or because the electrolyte is hydroscopic, it may have absorbed enough moisture to severely compromise the pH of the acid. The specimen that was protected with the chromium coating (Cr) provided approximately 150 hours of protection, and was followed by rapid cracking of the base material. The specimens protected with the electroplated nickel (Ni), electroplated nickel and chromium (Ni/Cr), and electroless nickel and chromium (E-Ni/Cr) all withstood the 300 hours of exposure with no degradation of the base material.

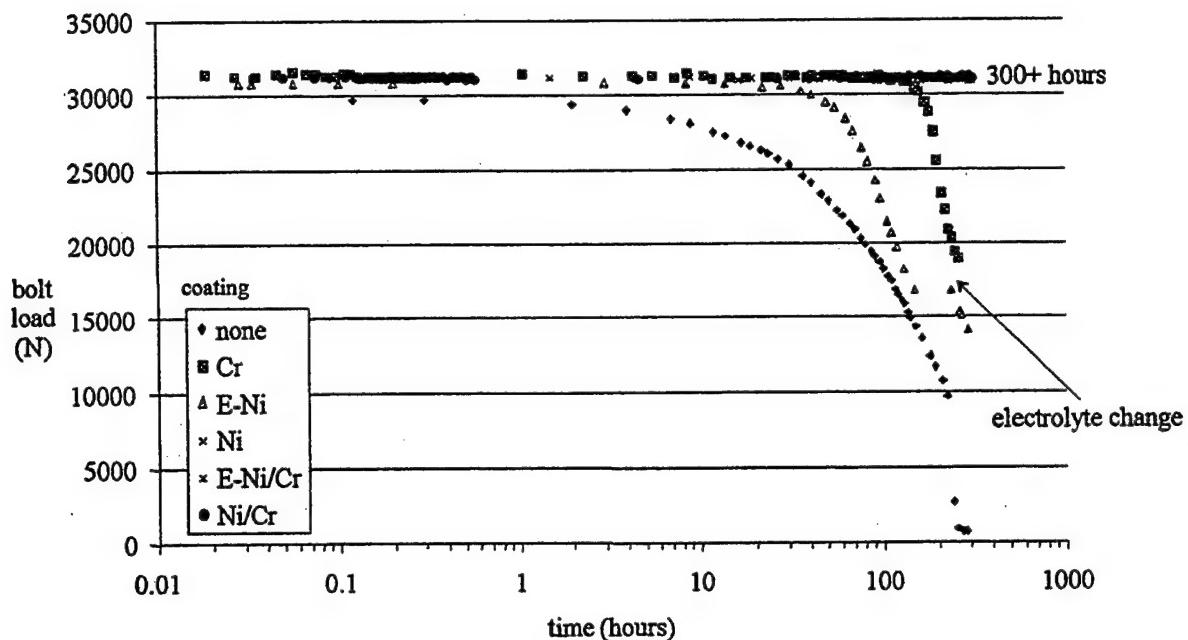


Figure 4. Environmental cracking resistance of Maraging 200.

A723

Figure 5 shows results of the tests on the 1300 MPa A723 steel. The first thing to note is that the A723 steel with no protective coating provided less than one hour of incubation time, and cracking had proceeded to the threshold in less than two hours. The chromium coated specimen provided approximately 0.1 hour of incubation time and had proceeded to the threshold in less than one hour. We believe this is a direct result of microcracking, which is often observed in chromium plating. This microcracking acts as crack initiation sites and rapidly accelerates the incubation time. The specimen coated with electroless nickel provided approximately 280 hours of incubation time, at which time the specimen began shedding load. At approximately 290 hours of exposure, the cracking arrested and an electrolyte change was performed. Cracking immediately resumed and at 300 hours, the specimen had attained the threshold. The specimens with electroplated nickel, electroplated nickel and chromium, and electroless nickel and chromium attained the 300 hours of exposure with no cracking and no shedding of load.

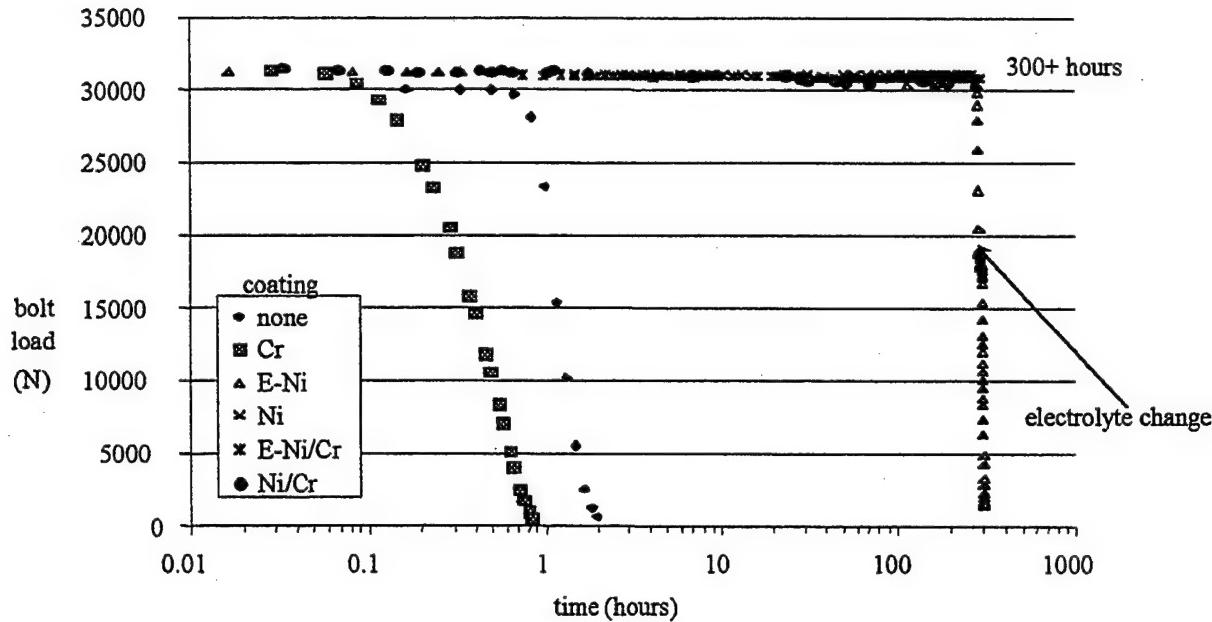


Figure 5. Environmental cracking resistance of A723 steel.

CLOSING REMARKS

1. Both Maraging 200 and A723 are highly susceptible to hydrogen-induced cracking. The three necessary conditions for hydrogen cracking are:
 - A source of hydrogen
 - A susceptible material
 - A sustained tensile loading
2. Because the sustained tensile stresses are difficult to prevent, the only logical solution to prevention of hydrogen cracking is to prevent the environment from reaching the high-stressed areas.
3. An electroplated nickel coating will prevent hydrogen from cracking susceptible materials such as high strength A723 steel and Maraging 200. The electroless nickel coating did increase the base material's resistance to hydrogen cracking. However, it was not as effective as the electroplated nickel.
4. Nickel may have problems adhering to some base materials, such as high alloy steels (e.g., Maraging 200); however, if the plating operations are properly performed, most low alloy steels (A723) can be easily plated.
5. Nickel is a relatively soft material and will need to be protected from abrasion with a hard material such as chromium plate. Nevertheless, plating chromium over nickel is a common practice and should pose no problem to a quality plater.

6. Chromium plating can provide protection to the underlying base materials if the plate is properly applied, and it is free from microcracks. Since this is difficult to obtain (as seen in the study here), we would not recommend it for environmental protection.

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